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EXTRACTION OF REACTIVE DYE USING NEW SUPPORTED LIQUID MEMBRANE

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Engineering (Chemical)

Faculty of Chemical Engineering
Universiti Teknologi Malaysia

MARCH 2014

To my beloved parents, younger brother and sister

ACKNOWLEDGEMENTS

Foremost, I would like to express my deepest appreciation to all those who provided me the possibility to complete this thesis. A special gratitude I give to my supervisor, Associate Professor Dr Norasikin Othman for the useful comments, remarks and guidance through the learning process of this master thesis. Without her guidance, this thesis would not have been possible. Secondly, I gratefully acknowledge my co supervisor, Prof Ani Idris for her assistance and guidance in my research.

Furthermore, I would like to thank to my fellow lab mates: Ooi Zing Yi, Raja Norimie, Nur Alina, Norul Fatiha and Norela for providing support, help and friendship that I need. I also like to thank to Siti Sabrina, Nur Syukriah and Siti Nadiah for their encouragement, friendship and being supportive throughout the research process.

Last but not least, I would like to offer my special thanks to family, En Harruddin, Pn. Rokiah, Satila Harruddin, Mohd Adam Afif and my fiance, Zafril Rizal for their understanding and supported me throughout entire process. I will be grateful forever for your love. To those who indirectly contributed in this research, your kindness means a lot to me. Thank you very much.

ABSTRACT

Currently, it is estimated that around 10,000 tons of dyes have been discharged around the world and causing some environmental problems. Since conventional treatments are not effective to degrade discharged dyes, efficient method should be applied to treat these pollutants. Supported liquid membrane (SLM) is an effective treatment for the removal of reactive dyes from wastewater because it provides maximum driving force for the separation of targeted solute and simultaneous extraction and stripping process, which lead to excellent separation. In this research, kerosene-salicyclic acid-tridodecylamine liquid membranes were used. Several factors that influence the stability of the SLM process, such as characteristics of the polymeric support and operating parameters were identified. The fabricated support was produced using thermally induced phase separation (TIPS). During fabrication process, different concentration of polymers at 10 wt%, 15 wt% and 20 wt% were tested. Several operating parameters for separation of Red 3BS reactive dye such as flow rate and pH of feed phase, concentration of stripping agent and concentration of feed phase were investigated using commercial support in order to find favourable process conditions. Results showed that the fabricated support with 15% of polymer concentration performed well as a membrane support with 100% of extraction and 58% of recovery of Red 3BS dye at favorable condition of 0.1 M sodium hydroxide as stripping agent, 100 ml/min of feed flow rate, 50 ppm Red 3BS and pH 3 of feed phase. The stability test also proved that the fabricated membrane remained stable up to 25 hours without suffering any breakage on its structure. As a conclusion, the fabricated support was proven to have high potential as a membrane support due to its high stability and excellent performance in separation process.

ABSTRAK

Pada masa kini, dianggarkan sekitar 10,000 tan pewarna dibuang di seluruh dunia dan menyebabkan beberapa masalah alam sekitar. Memandangkan kaedah lazim tidak efektif untuk mendegradasi pewarna yang dibuang ini, kaedah yang berkesan patut diaplikasi untuk merawat pencemar ini. Membran cecair bersokong merupakan kaedah yang efektif untuk penyingkiran pewarna reaktif dari air sisa kerana ia membekalkan daya pacu yang maksimum untuk pemisahan bahan larut yang dikehendaki dan proses pengestrakkan dan pelucutan yang berlaku secara serentak yang membawa kepada proses pemisahan yang cemerlang. Dalam kajian ini, membran cecair kerosin-asid salisiklik-tridodesilamina telah digunakan. Beberapa faktor yang mempengaruhi kestabilan membran cecair bersokong, seperti ciri-ciri penyokong polimer dan parameter operasi telah dikenalpasti. Penyokong fabrikasi telah dihasilkan menggunakan kaedah pemisahan fasa didorong terma (TIPS). Semasa proses fabrikasi, kepekatan polimer yang berbeza pada 10 wt%, 15 wt% dan 20 wt % telah diuji. Beberapa parameter operasi untuk pemisahan pewarna reaktif Red 3BS seperti kadar alir dan pH fasa suapan, kepekatan agen pelucutan, dan kepekatan larutan fasa suapan telah dikaji dengan menggunakan penyokong komersil untuk mendapatkan keadaan proses yang sesuai. Keputusan menunjukkan penyokong yang difabrikasi dengan kepekatan 15% polimer menunjukkan prestasi yang baik sebagai penyokong membran dengan 100% pengekstrakkan dan 58% perolehan semula pewarna Red 3BS pada keadaan yang sesuai iaitu pada 0.1 M natrium hidroksida sebagai agen pelucutan, kadar aliran suapan 100 ml/min, kepekatan Red 3BS 50 ppm dan fasa suapan pH 3. Ujian kestabilan juga membuktikan membran fabrikasi kekal stabil sehingga 25 jam tanpa mengalami sebarang pemecahan pada strukturnya. Kesimpulannya, penyokong yang difabrikasi ini terbukti berpotensi sebagai penyokong membran kerana mempunyai kestabilan yang tinggi dan prestasi yang cemerlang dalam proses pemisahan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

LM	-	Liquid Membrane
SLM	-	Supported Liquid Membrane
ELM	-	Emulsion Liquid Membrane
BLM	-	Bulk Liquid membrane
TIPS	-	Thermal Induced Phase separation
iPP	-	Isotactic Polypropylene
DPE	-	Diphenylether
SA	-	Salicyclic Acid
TDA	-	Tridodecylamine

LIST OF SYMBOLS

w_o	-	weight of wet membrane (g)
w_i	-	weight of dry membrane (g)
A	-	membrane area (cm^2)
h	-	membrane thickness (cm)
V_{pores}	-	volume of pores membrane (cm^3)
V_{total}	-	volume of total membrane (cm^3)
C	-	concentration of Red 3BS at given time (ppm)
P	-	permeability of membrane (cm^3/cm^2.min)
Ae	-	membrane area in contact with aqueous phase (cm^3)
ε	-	porosity of the membrane material
J	-	flux value (mol/cm^2.min)
D	-	diffusion coefficient of the complex (cm^2/min)
L	-	membrane thickness (cm)
C_{fi}	-	concentrations of solute at the feed/membrane interface (ppm)
C_{si}	-	concentrations of solute at the membrane/strip interface (ppm)
kf	-	rate constants forward reaction($M.s^{-1}$)
kr	-	rate constant reversible reaction ($M.s^{-1}$)

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Nowadays, awareness of society towards protection of environment has tremendously increases. Water pollution is a type of pollution that caused major destruction to the environment especially to river, lakes, ocean and underground water (Muthuraman and Palanivelu, 2006a; Brown and Devito, 1993; Turgay *et al*., 2011). Increasing human population and industrial developments have caused rise in the amount of wastewater discharged to the environment. Wastewater irrigation poses several potential threats to the environment and human health via the contamination and exposure of pathogenic microorganisms, heavy metals and harmful organic chemicals causing lack of suitable water for drinking, agriculture and farming industry (Stagnitti, 1999). Besides, variety of pathogens found in wastewater including bacteria, protozoans and virus are dissociated with several infections such as diarrhea, vomiting, typhoid and malabsorption, which caused serious diseases to human.

Wastewater produced from textile industries is the significant water pollution source (Solozhenko *et al*., 1995; Marechal *et al*., 2012). Every year, it is estimated that over 10,000 tons of wastewater from textile and dyeing is produced and require efficient treatment before being discharged to water source (Forgacs *et al*., 2004). Textile wastewater contains a large number of harmful chemical such as formaldehyde, dioxin, reactive dyes, pesticides, halogenated benzene, organic chemicals, biocides, surfactants and toxic organic chemicals (Hussein, 2011;

Marechal *et al*., 2012). Reactive dye is one of the major components found in wastewater that exhibit as colored wastewater and resistant to any degradation process. During dyeing process, approximately 75% of dyes were discharged by Western European textile processing industries and 36% of it belonged to reactive dyes (Andrea, 2005). Reactive dye is mainly used for coloration of cotton and cellulosic fiber and currently represents 20 - 30% of the total market share for dyes (He *et al*., 2010). This component is extensively used as colorant due to their high degree chemical, photolytic stability, and high ability to bind with textile fibers through the covalent bonds. Therefore, strong adhesion between reactive dye and fiber is formed and render it hard to be removed from washing process (He *et al*., 2010; Hassan *et al*., 2009; Tekoglu and Ozdemir, 2010). Although reactive dyes have good interaction with the fabric, but it poses various negative impacts on environment and human being. Wastewater from dyeing stage commonly presents as toxic, mutagenic and carcinogenic effect on aquatic life (Lu *et al*., 2010; Banat *et al*., 2006). Discharging dye-containing wastewater to water effluent can cause great damage to human body, reproductive system, function of kidney, liver, brain and nervous system (Kadirvelu *et al*., 2003). Moreover, this component exhibits high integrity and resistance towards microbial degradation, chemical, thermal and photolytic degradation due to their complex chemical structure. As a consequence, the accumulation of dye components into the water bodies causes severe impact to human community and direct destruction to aquatic community. Therefore, it is crucial to find an effective method to treat the reactive dye wastewater. However, process to degrade and treat reactive dyes is difficult because generally they are stable in light, resistant to aerobic digestion, complicated in structure and not biologically degradable (Kumar *et al*., 2011).

A wide range of wastewater treatment has been developed such as oxidation-ozonation (Arslan and Balcioglu, 2006; El-Desoky *et al*., 2010), coagulation electrochemical techniques (Soloman *et al*., 2009; Klimiuk *et al*., 1999), biological treatment (Ranjusha *et al*., 2010; Turgay *et al*., 2011), adsorption (Donnaperna *et al*., 2009), foam fractionation (Lu *et al*., 2010), photocatalytic degradation (Attia *et al*., 2008) and membrane separation process (Fogarassy *et al*., 2009; Sostar-Turk *et al*., 2005). Conventional treatments usually involve physical, chemical and biological treatments for refining the textile wastewater. However, conventional treatment is proven non- effective for handling textile wastewater due to the chemical stability of these dye pollutants towards any degradation process. Reactive dyes have high degree chemical, high solubility and strong complex aromatic molecular structure resulting in greater difficulty in degradation and removal from water source (Ding *et al*., 2010). These characteristics of dyes affect the effectiveness of treatment of wastewater. Existing conventional treatments poses various disadvantages such as sludge generation, formation of byproducts, short-half life, high cost operation, release of aromatic amines, long time of process and not effective for all type of dyes (Robinson *et al*., 2003; Ahmad *et al*., 2010). Therefore, a new approach of separation based membrane technology, which is supported liquid membrane was applied and proven as the best alternative treatment for removal of dyeing components in wastewater and can be applied for large scale ecologically friendly treatment process (Wang *et al*., 2011).

Supported liquid membrane (SLM) is part of membrane technology that shows a great potential in the separation of desired solute from an aqueous phase since it combines extraction and stripping process in one single step unit operation. The one single step process provides the maximum driving force for separation which leads to an excellent removal and recovery process (Ho *et al*., 2001; de Agreda *et al*., 2011). Potential advantages of SLM treatment over the conventional treatment are small amount of organic phase and carrier, high separation factor, high selectivity, minimal amount of carrier, low operating capital, and operating cost and easy to scale up (Mahmoud *et al*., 2007; Fu *et al*., 2003). The use of SLM to remove and recover dye ions from wastewater has long been pursued by researcher community such as Muthuraman and Palanivelu (2006b), Hajarabeevi *et al*. (2009) and Nisola *et al.* (2010). This treatment shows great potential in removal and recovery of dye ions from aqueous solution due to their non-equilibrium mass transfer characteristics and very low inventory of organic solvent. Previous research done by Muthuraman and Palanivelu (2006) and Hajarabeevi *et al.* (2009) showed that the maximum extraction and recovery of dye ions were achieved by using SLM process under optimum conditions. Thus, this treatment has been proven attractive due to an excellent performance in separation and recovery of desired solute.

1.2 Problem Statement

Reactive dyes are mainly used in textile industry due to favourable characteristics of bright colour, water-fastness and formation of strong interaction with the fabric. These kinds of characteristics are due to their complex chemical structure by having benzidine ring substituent of azo coupling to aromatic system and presence of other aromatic amine compounds (Mathur *et al.*, 2012). These compounds retain color on fabric and along with high integrity. However, presence of reactive dyes in wastewater causes destruction to mankind and environment. As a consequence, the release of dye into the water bodies causes severe impact to human community and direct destruction to aquatic community. Supported liquid membrane process is demonstrated to have significant potential as an effective tool for separation of reactive dye from textile wastewater. This treatment performs high separation efficiency and selectivity towards desired solute (Bukhari *et al*., 2004).

Despite many advantages, this technology has rarely been applied in industry due to certain limitation such as instability and short lifetime of SLM process. The major reason for this is membrane support stability and lifetime. Instability phenomenon occurs when the liquid membrane fails to retain in the pore of membrane support leading to carrier lost (Calzado *et al*., 2001; Arslan *et al*., 2009). This problem has an influence on the flux and selectivity of SLM process. Factors influencing the stability and lifetime of SLM process have been studied extensively by previous research (Yang and Fane, 1999; Zheng *et al*., 2009a). There are many factors that influence the stability of SLM such as type and characteristic of polymer support, membrane solvent, carrier and operating temperature. Membrane support is one of the factor that greatly influenced the stability and performance SLM process (Dżygiel and Wieczorek, 2010). Since the liquid membrane is held within the pores of support by capillary force, it is clear that the pore structure, morphology and porosity influenced the stability of process. The support should have porous structure to ensure the continuity the transportation of solute throughout the support. Besides, it is found that symmetric support is suitable for SLM process because it has fixed pores structure throughout the support and interconnected with each other. Suitable morphology of support is a promising efficient separation of reactive dye with high stability and lifetime of process.

Supported liquid membrane stability and lifetime limit the industrial applications of this separation technique. Therefore, the stability of the support needs to enhance drastically. A proper choice of method of fabrication support and membrane support composition might improve the lifetime SLM system. A new approach is taken for enhancing the stabilization of SLM process by fabricating a support using thermally induced phase separation (TIPS) technique. TIPS is well known because it is a versatile and simplest technique for producing porous support and it is believed to have the ability to produce support with suitable morphology for SLM process (Fu *et al*., 2003). TIPS technique has been widely used by previous researchers to develop microporous support for separation process (Yave *et al*., 2005; Matsuyama *et al*., 2002b; Li *et al*., 2006). Previous studies by Fu *et al.* (2003) and Fu *et al*. (2004) have fabricated the support that specific for SLM process and it is proven to be effective in separation of solute. Therefore, it is believed that these new approaches have high capability in improving the performance of separation and enhancing the stability of SLM process. Several improvement and modification in TIPS technique have been proposed in order to achieve a maximum separation and stability of SLM process.

1.3 Objectives of Study

The main purpose of this study is to fabricate a support for separation of reactive dye in SLM process. In order to successfully attain this objective, there are several parameters in highlight for optimum extraction performance. The following are the objectives of this research:

i. To synthesize the fabricated support for reactive dye removal using TIPS technique.

ii. To investigate the favorable conditions for removal and recovery of reactive dye using commercial support.

iii. To study the performance of fabricated membrane support in extraction of reactive dye in SLM process.

1.4 Research Scopes

This study is to investigate the separation of reactive dye using fabricated membrane support in SLM process. To achieve the objective of this study, fabricated support was produced and identification of favorable condition for separation of reactive dye using commercial membrane support was investigated.

For fabrication of support, isotactic polypropylene (iPP) was used as a polymer and diphenylether (DPE) was used as a diluent. TIPS technique was chosen as a method of fabrication support due to easiness in controlling membrane structure. It is believed that TIPS technique is feasible for controlling structure and pore of support by inducing different concentration polymer-diluent. Three different concentration of polymer-diluent (10% iPP-90% DPE, 15% iPP-85% DPE, 20% iPP-80% DPE) were studied. The physical characteristic and morphology of fabricated support was measured by using Scanning Electron Microscopy (SEM). From the SEM analysis, structure of cross section and pore size membrane were illustrated.

In order to test the performance of fabricated support, the favorable condition of SLM should be studied. The commercial support was used as a support for liquid membrane. Reactive dye Red 3BS was used as a feed phase in this study. For organic phase, tridodecylamine (TDA) was used as a carrier assisted by salicyclic acid (SA) as a co-carrier with kerosene as a diluent (Othman *et al*., 2011). At the stripping side, sodium hydroxide was used as a stripping agent. In order to find the best operating condition for SLM process, several parameters processes were attempted such as flow-rate of feed phase (50 ml/min, 100 ml/min, 125 ml/min, 150 ml/min), pH of feed phase (pH 1, pH 2, pH 3, pH 4, pH 7.07), concentration of stripping agent (0.25 M, 0.5 M, 0.1 M, 0.2 M) and concentration of feed phase (10 ppm, 30 ppm, 50 ppm, 70 ppm). The performance of removal and recovery of reactive dye was analyzed by UV spectrophotometer.

The performance of fabricated support was studied using SLM process at favorable condition obtained in the second objective. Fabricated support with different polymer concentrations was tested in SLM process. Different morphology of fabricated support led to different performance on separation of reactive dye. For each process, 6 hours of operation was needed to completely perform the removal and recovery of reactive dye from an aqueous solution. At the end, the stability and lifetime of fabricated membrane support was studied by allowing the separation process continuously without re-impregnation of fabricated membrane support in liquid membrane until it showed some instability behavior. Concentration of feed and strip phase were analyzed every 30 minutes by UV spectrophotometer.

1.5 Research Outline

This thesis consists of five chapters, which present the research in sequential order. Chapter 1 introduces the research background, problem statement, objective of study and research scope. Besides, the research outline of the thesis is also included in this chapter. Chapter 2 represents the detailed reviews those related with textile industry and wastewater treatment, liquid membrane technology, application of SLM process, fabrication of support using TIPS process and future development of SLM process. Chapter 3 describes the materials used and methodology involved in this study. Experimental procedures include fabrication support using TIPS method and separation of reactive dye using SLM system was discussed in detail in this chapter. In order to achieve the objectives, the scope of works including fabrication of support using TIPS method, identification of favorable condition of SLM process, capability of fabricated support as a membrane support for separation of Red 3BS and the stability of commercial and fabricated membranes support were investigated. In addition, the morphological structure and pore size of fabricated support was analyzed using SEM test. The results and discussions are presented in Chapter 4. In this chapter, experimental data collections were been discussed and analyzed in detailed. Finally, the conclusion and recommendation are presented in Chapter 5 for further improvement in future research.

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